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FEATURE ARTICLE

HIGH PRECISION CRYSTAL ORIENTATION MEASUREMENTS WITH THE X-RAY OMEGA-SCAN - A TOOL FOR THE INDUSTRIAL USE OF QUARTZ AND OTHER CRYSTALS

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The X-ray Omega-Scan method has been developed in order to get the single crystal orientation with very high precision (± 0.001^0). It is shown how this method helped to improve the quartz oscillator production all around the world. The application of the Omega – Scan method for other crystals as e.g. sapphire and silicon carbide, is described. It has been firmly established that the method is suitable for arbitrary single crystals with arbitrary lattice geometry and symmetry.

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1. Introduction

One of the most prominent properties of quartz crystals, their piezoelectricity, had been discovered already in 1880 by the brothers Pierre and Jacques Curie. A practical application of this effect on a larger scale, however, started much later: Only for about sixty years quartz is now in use as the basic material of electronic oscillators.

Originally one had to rely on naturally grown quartz crystals of high quality. But due to the increasing need for oscillators the sources for this material nearly ran out and its price drastically increased. However in the meantime one had learned to grow nearly perfect quartz artificially in large vessels at elevated pressures and temperatures. The results are quartz „stones“, which are cut for the final configuration into small slices, the „blanks“. Covered with gold or silver films the blanks are ready to be contacted and mounted in evacuated or gas-filled housings.

As an important quality of an oscillator its frequency has to be nearly independent of thermal changes in a not too narrow temperature range. This is true only for certain orientations of the blank's surface relative to the quartz lattice which must be realized with an accuracy of a few seconds of arc. Usually the quality of the cutting process (sawing, grinding) is not sufficient to meet these requirements. The dependence of the oscillator frequency on the temperature range is measured in thermostates, and originally the result was a loss of up to 50% of the completed products.
About a dozen years ago, however, it has been shown [1] that miscuts can be detected in a much earlier production step by applying methods of X-ray diffraction, reducing production costs in this way.

As is well-known, X-ray diffraction in crystals had been detected in 1912 by Max Laue in Munich, experimentally supported by W. Friedrich and P. Knipping ([2] reprinted in [3]). This was considered as a proof for the wave nature of X-rays (at that time) and the existence of space lattices in crystals. Shortly later the Braggs (Sir William Henry, the father, and Sir William Lawrence, the son) contributed the „Bragg equation“ [4], leading to an easier understanding of what was then called the

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„kinematical theory of X-ray diffraction“, valid for imperfect crystals. All three of them were honoured with Nobel Prizes, M.v. Laue in 1914, the Braggs in 1915. A dozen years later Max von Laue completed the theoretical base of X-ray diffraction by establishing the „dynamical theory“, valid for perfect crystals.

After World War II M.v. Laue at age 72 was appointed director of the oldest institute of the former Kaiser-Wilhelm-Gesellschaft, the „Kaiser-Wilhelm-Institut für Physikalische und Elektrochemie“ in Berlin-Dahlem, to which Laue shortly later gave the name Fritz-Haber-Institute. Both authors of the present article have been employees of Laue’s institute for a shorter or longer time, H.B. in Prof. Hosemann’s and G.H. in Prof. Borrmann’s department.

In the following two Figs. 1,2 Prof. v. Laue can be seen at the blackboard of the (no longer existing) „small auditorium“, celebrating with a lecture the 50th anniversary of his doctorate, and at the entrance of his institute shortly before his unexpected death (1960) caused by a traffic accident. For more details cp. [5].

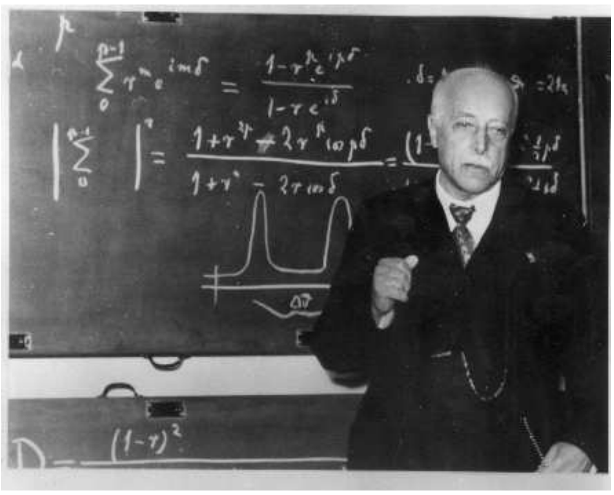


Fig. 1. Professor Max von Laue on the occasion of his 50th doctor anniversary.

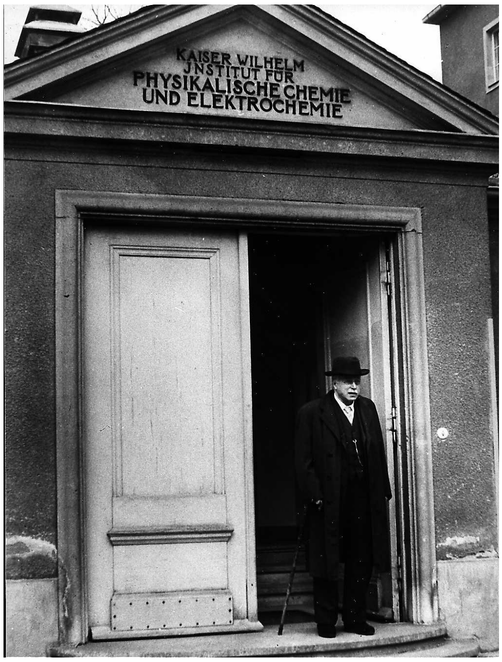


Fig. 2. Professor Max von Laue at the entrance of the Fritz-Haber Institute.

In the following Chapters we will learn in which way X-ray diffraction has helped to improve the quartz oscillator production.

2. The quartz material - growth and properties

2.1. Growing of the “stones“

As already mentioned in the Introduction, the industrial application of quartz started with the use of naturally grown crystals which in the required quality were present only at certain places, e.g. in the Ural Mountains. Due to the rapidly increasing need these mineral resources are nearly exhausted nowadays with the consequence of extremely high prices (about one thousand US$ per kilogram).

Much cheaper are α-quartz crystals artificially grown on oriented seed crystals in huge vessels (steel autoclaves with diameters up to 0.5 m and heights up to 8 m, see Fig. 3).

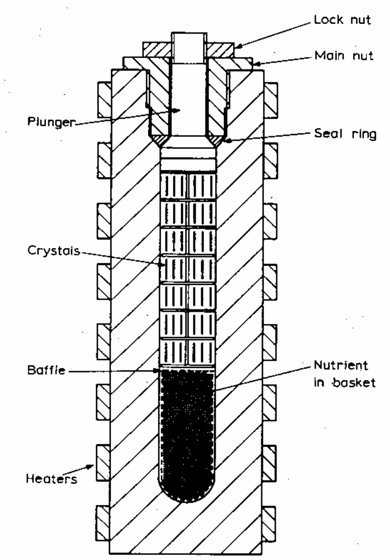


Fig. 3. Autoclave for the growth of quartz crystals. - From [6].

Fed by a nutrient consisting e.g. of natural quartz chips + water + NaOH, the hydrothermal quartz crystals grow at temperatures of 400 °C and pressures above 10^8 Pa with rates 1...2 mm per day to “stones“ with measures of e.g. 300 × 150 × 50 mm^3 within a few weeks. These crystals are relatively free of defects, depending partly on the perfection of the seed crystals. The seed in the center of such a stone is visible in Fig. 4a, whereas in Fig. 4b (same stone seen from above) the metallic cramps fixing the seed are to be seen on the left side.

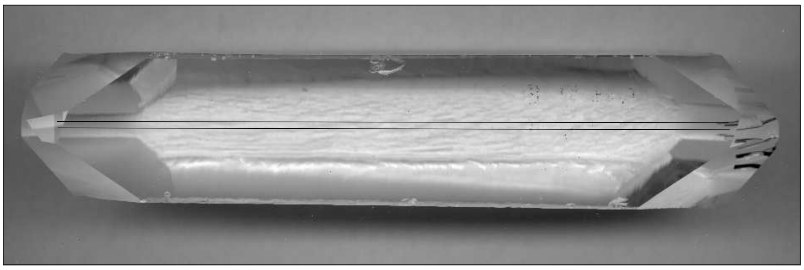


Fig. 4a. Stone with seed plate.

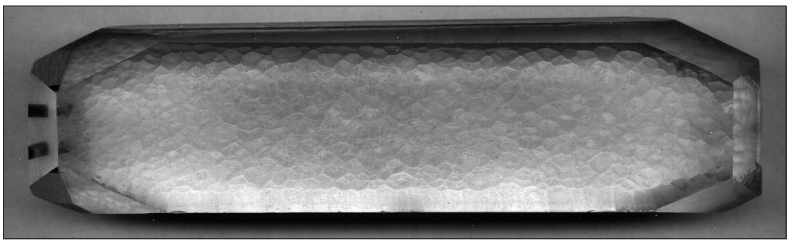


Fig. 4b. Same stone, seen from above.

Still much more difficult is the growing of sufficiently large and perfect crystals from other piezoelectric materials (berlinite, galliumphosphate, the langasite family). These materials, however, have partly superior properties, allowing for filters with larger band widths and frequency shifts. For more details see [7].

2.2. Defects in hydrothermal quartz crystals

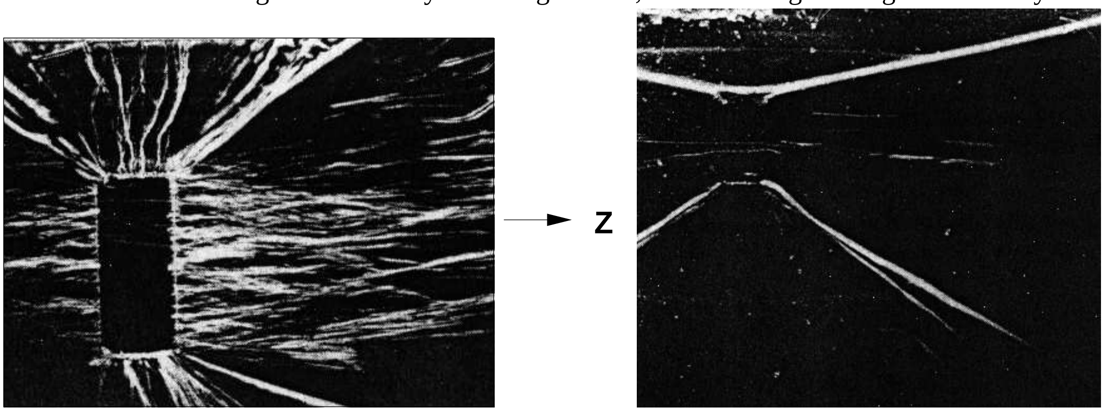
Almost all synthetic quartz crystals have a veil of inclusions around the seed plate resulting from initial rapid growth and originally full of solvent. This veil region, sometimes obvious to the naked eye, should never be used for device production. It often implies a high dislocation density. Most dislocations originate from veil inclusions or are continuations of dislocations already present in the seed crystal. With selected dislocation-free seed plates stones with a nearly perfect lattice can be grown.

Dangerous is a high hydrogen content: it makes the dislocations mobile and able to multiply.

The detection of dislocations can be performed by using the well-known methods of X-ray topography, cp. Figs. 5a, b. These methods have also made clear, that inclusions, if present, nucleate at least one dislocation.

Because a correlation between hydrogen content [H] and the dislocation density N_D has been confirmed, industry prefers to determine the H content by measuring the infrared absorption. One then concludes on the dislocation density assuming the validity of $N_D \sim [H]^{2.5}$.

Actually a small number of dislocations in quartz oscillator plates can be tolerated. Dangerous, however, is every change in the dislocation density, because it can change the resonance frequency of the oscillator. Such changes occur slowly after longer times, such lowering the long term stability.



(a) (b)

Fig. 5. X-ray topographies from artificial quartz crystals. From [6]. (a) In a relatively "bad" crystal many dislocations originate from the veil around the seed. (b) In a more perfect crystal only a few dislocations are visible.

2.3. Mechanical treatment

As the first step of oscillator production the so-called „lumbered bars" are cut out from the stones (mentioned in section 2.1.) in appropriate directions, e.g. in the direction of the Y axis of the quartz lattice and with side faces vertical to the X and Z axes (the positions of the axes and of two industrially used cuts are indicated in Fig. 6).

Sometimes also other orientations are used for special purposes, see below.

Quartz has a hardness comparable to that of glass, so the cutting cannot be done just by simple sawing. Instead one takes quartz crystals apart using diamond-coated discs (they are available as inboard or outboard diamond saws, resp.), or one grinds them using steel bands or grinding wires, working together with a grinding liquid (water + corundum powder).

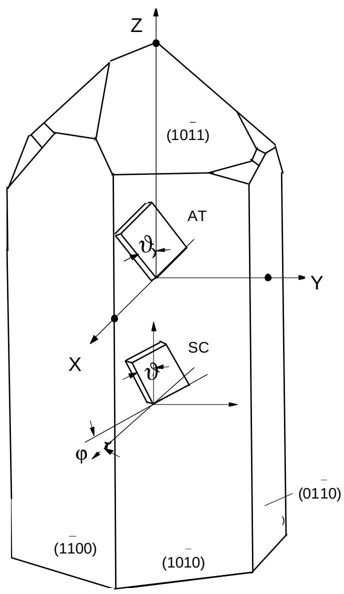


Fig. 6. Shape of a naturally grown quartz crystal. No natural faces exist vertical to the X- and (optical) Z-axis. The positions of two important cuts are indicated (see below in the text). Their angles φ against the X - axis and ϑ against the Z - axis are approximately:

AT-cut $\varphi = 0^0$, $\vartheta = 35^0 15'$; and SC-cut: $\varphi = 23^0$, $\vartheta = 34^0$.

Similar methods are applied to prepare small quartz discs from the lumbered bars, which industry calls „blanks“. Usually they have measures 15 mm ....1 mm and thicknesses 100 µm ....10 µm. For other purposes „wafers“ are produced as an intermediate product, larger discs with measures e.g. 15×65 mm^2 and 300 ...20 µm thickness. Oftenly blade saws are in use (Fig. 7), consisting of up to 100 strongly tight parallel steel bands wetted with the above mentioned grinding liquid. One sawing run takes a few hours, and because the steel bands wear very quickly they have to be replaced many times by new ones after time-consuming adjustment.

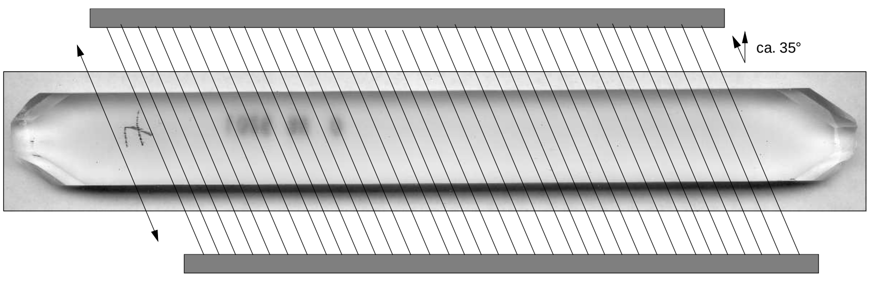


Fig. 7. Principle of a blade saw for cutting raw AT-blanks from a pre-oriented lumbered Y-bar.

After cutting the blanks one grinds or cuts them, if necessary, to the desired lateral dimensions. This holds especially for round blanks, which are mostly in use for geometrical reasons. By glueing the rectangular discs together to a long stick and grinding it to a round shape one gets round blanks after dissolving the glue.

The half-finished blanks are then lapped to nearly the desired thickness: The blanks are placed in plastic carriers between upper and lower metal laps which are fed with an abrasive slurry. Most manufacturers regard the details of the lapping process as trade secrets. In any case the lapping process damages the surface. Since such a damage is undesirable it is removed either by using successively finer abrasives finishing or by etching. However polishing to a visually satisfactory finish does not guarantee removal of all surface strain. But etching again is a complex technology. One problem are the dislocations: Most etchants preferentially attack the quartz around dislocations with the possible result of „etch tunnels“, channels linking both faces of a blank. This problem can be serious when thin blanks are needed.

3. The cutting angles

The cutting angle of the blanks, i.e. the orientation of the surface of an oscillator plate relative to certain quartz lattice planes or axes, is of highest importance. Many properties of the final product, e.g. the insensitivity against the temperature fluctuations or accelerations (satellites!), the long term stability etc. depend strongly on the correct keeping of certain surface orientations.

Still mostly used (and produced at rates of millions per week) are blanks with the so-called “AT-cut“, see Figs. 6, 7, 8 (Fig. 8 shows the projections of the normals of 1/12 of the quartz lattice planes onto a sphere, together with the projections belonging to the most prominent blank surface positions like AT, SC and others). The AT plane is inclined against the (strongly X-ray-reflecting) lattice plane $011 \equiv (01\bar{1}1)$ by nearly 3°, a fact opening up the possibility for an easy control procedure using X-rays, see section 5.

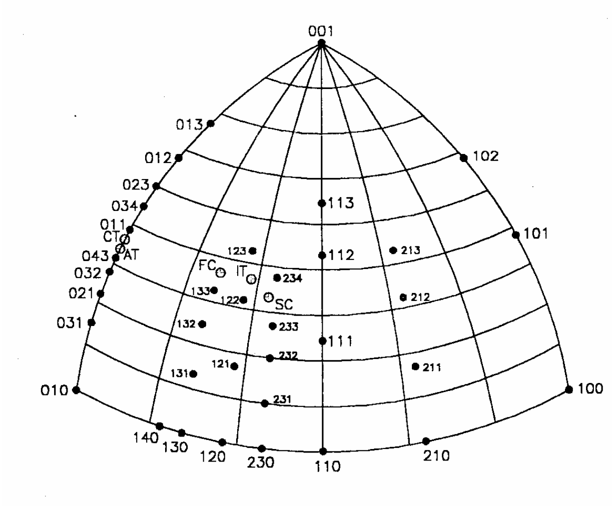


Fig. 8. Projection of the normals of 1/12 of the most important quartz lattice planes and of some cuts onto a sphere. Projection 001 belongs to the Z- (optical) axis, no related plane; 010 belongs to the Y-axis (plane $(01\bar{1}0)$). - From [7].

As an example for the temperature behaviour of AT cut blanks Fig. 9 shows the frequency deviations ΔF (in parts per million) in an temperature interval between -75 and +125°C (each adjacent curve belongs to a change of 2 arcmin in the surface position).

As has already been mentioned the AT position is very close to the $(01\bar{1}1)$ plane of the quartz lattice, see Fig. 8. In many cases one chooses $\varepsilon = 2°58'$ as the deviation from this plane. Since

$(01\bar{1}1)$ encloses an angle of 38°13’ with the Z axis one has to adjust the blade saws at an angle 38°13’ - 2°58’ = 35°15’ about the X-axis from the XZ plane, see Figs. 7, 8: one simple rotation is sufficient to arrive at the exact AT position.

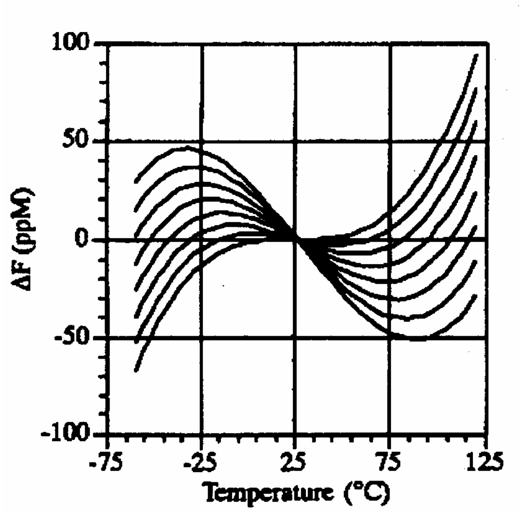


Fig. 9. AT-cut, frequency/temperature characteristics, depending on the actual cutting angle (angular difference 2 arcmin between neighbouring curves). - From Pond et al., 13th Piezoel. Dev. Conf. (1991) 93-100.

The AT-cut family of devices is and will continue to be very useful, cp. in watches, but it has some disadvantages: it is very sensitive to strains due to thermal and mechanical stresses. Other cuts than the AT one offer superior properties. In particular, in SC-cut devices strain does not affect the frequency. Therefore this cut comes more and more into use nowadays. As is clear from Figs. 6, 8 this cut (and others like IT, FC ...) cannot be adjusted by one rotation about the X-axis: a succeeding independent second rotation about another axis is necessary (Figs. 6, 10). Therefore, this family of cuts got the name “doubly rotated cuts“.

For the production of doubly rotated blanks industry uses pre-adjusted bars which already consider the first rotation. Then the same blade-saw equipment as used for the AT-production can be applied, with the saws now adjusted according to the second rotation. As a consequence of this complicated procedure rejects are here much more frequent.

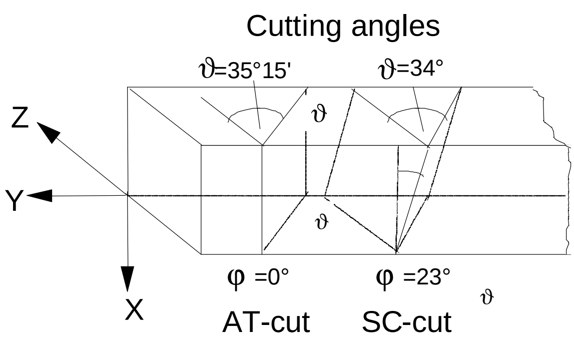


Fig. 10. AT-cut and SC-cut positions within a Y-bar. Left hand side: the AT-cut position is adjusted after one rotation about angle ϑ. Right hand side: adjustment of the SC-cut requires an additional rotation about angle φ.

Recently constructed X-ray controlled SC-cutting machines offer two advantages: they deliver precisely adjusted blanks meeting extreme demands, and they need the same bars as used for the AT cutting process. As a disadvantage, they work much slower due to a piece-by-piece production.

4. Final treatment

After the final surface treatment an X-ray control rejects blanks with inadequate surface orientations; this will be discussed below in much detail. The surfaces are then coated with thin silver films applying evaporation (gold is used only in high-quality devices). In a first step small basecoats applied to the blanks' faces serve as electrical connections, and in the second step the whole central regions of both oscillator faces are covered with a circular coat. In a third step and with the device now operating, the deposition of some additional silver (or gold) in the centre is stopped when the frequency reaches the target value. The quartz plate is then held in some mount serving as a mechanical holder and providing the electrical connection. The type of mount depends on the application of the device, and again there exist typical problems. Finally, the device is scaled in a case made of glass, ceramic, or metal. The final stages of treatment are of importance. For example, the long-term stability is increased if the components are baked in vacuum before use. Also the ambient in the enclosure is important. With a high vacuum best results are obtained, but dry nitrogen, hydrogen, or helium are also used. It takes a longer time that the blanks heats to its operating temperature in vacuum than in gases. To keep this temperature constant thermostats are used as well in the case of extremely high demands. Finally in most cases the temperature behaviour of each blank is checked.

5. Application of X-ray methods

X-ray methods are applied mainly to check two properties of quartz oscillator crystals: 1) to control the quality of the crystal lattice, 2) to measure the correct orientation of the blank surface with highest precision.

5.1. X-ray topography

A quality control can be performed using different methods of X-ray topography. Already simple reflection topography (crystal adjusted in the Bragg case) along the surface of seed plates, stones, extended wafers etc. gives hints about the perfection of the material, as shown in Fig. 11. More information provide transmission topographies (crystal adjusted in the Laue case), see Figs. 5a, b. Much easier (and thus mostly preferred by industry) is the measurement of a "quality factor" Q for getting a rough idea about existing dislocation densities via the hydrogen content, see section 2.2.

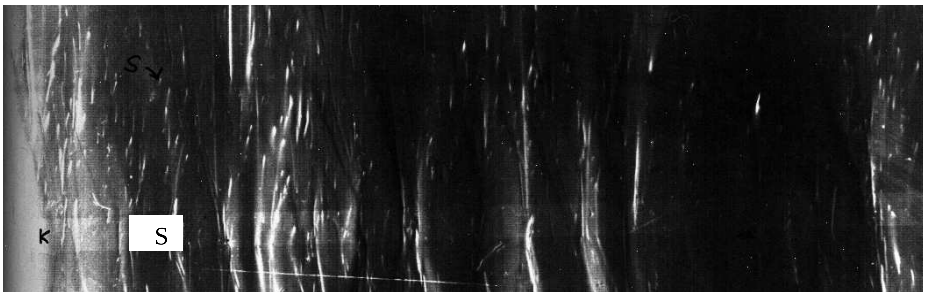


Fig. 11. Quartz surface (Lang-) topography, taken from a stone in reflection $04\bar{4}0$ with CuK_{α} radiation (from S. Moré, Diploma work 1992). In the lower part the seed plate is faintly visible (marked with S).

5.2. The Theta-Scan method

As mentioned above, the blanks must have their surface parallel to particular orientations with extremely high accuracy in order to keep frequency deviations as small as possible in certain temperature ranges. Deviations of ± 10 ppm (parts in 10^6) over a range of -20 °C ... +70 °C are usually adequate, but professional telecommunication (and military applications) require less than 1 ppm. For satellite navigation even deviations of <0.01 ppm are called for. Plate orientations meeting these requirements can be measured only applying X-ray methods.

For a long time the so-called Theta-Scan method was the only method to allow measurements with this extreme accuracy. In an adequate arrangement (still used nowadays at some places and for certain purposes) an AT-blank, just to give an example, is slowly scanned through the Bragg position, which in this case results from the reflection of X-rays on the $(01\bar{1}1)$ lattice plane according to the Bragg law

$$n \cdot \lambda = 2d_{01\bar{1}1} \sin\Theta_B$$

(λ is the X-ray wavelength, n is the order of diffraction, d is the lattice parameter, Θ_B is the Bragg angle of diffraction). If the incident beam is adjusted to a fixed angle $\Theta_B + \varepsilon$ against the blank surface then the position of the surface against the $(01\bar{1}1)$ plane is determined by exactly measuring the angle ε, see Fig. 12.

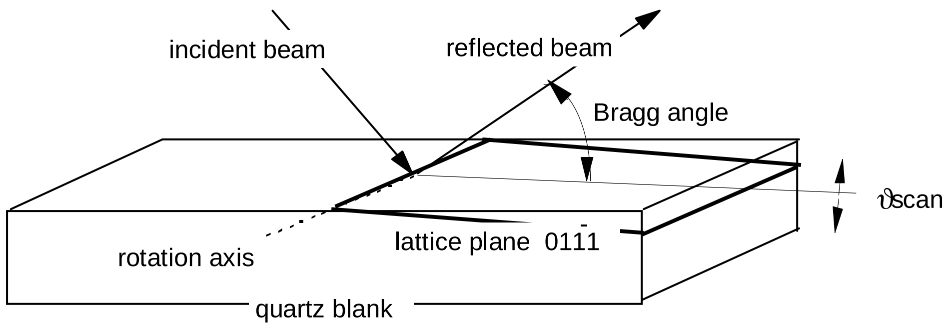


Fig. 12. Θ-Scan arrangement. Incident and reflected beams are in fixed positions, the AT-blank is rotated about the indicated axis, which is parallel to crystal axis X. The lattice plane is inclined to the blank surface at an angle ε.

The Θ-Scan method is mainly restricted to the measurement of rectangular blanks (and is simple only in the case of AT-cut blanks). But its principally high reproducibility (down to 1 arcsec) is misleading if the $(01\bar{1}1)$ plane is not in symmetric position within the blank (causing the so-called XX'-error) or if the blank edges are not correctly aligned. It is, however, successfully applied for the automatic adjustment of lumbered quartz bars for the succeeding cutting of AT-cut blanks. Significant problems arise with measurements of round blanks, as well as with doubly rotated blanks, where angles against two lattice planes have to be considered.

5.3. The Omega-Scan method

The Omega-Scan, presented about a dozen years ago [1] and improved in many details since that time [8] is able to overcome the largest part of the just mentioned problems and to fulfil the new greater demands.

In an Ω-Scan apparatus the (round) blank is deposited on a rotating turntable, see Fig. 13. The narrow focused incident X-ray beam is fixed in such a direction that in the case of an AT-cut the Bragg condition on $(01\bar{1}1)$ is not fulfilled in the plane of symmetry: if ε is the angle between $(01\bar{1}1)$ and the surface of the blank, the angle of incidence is not adjusted to $\Theta_B + \varepsilon$, but somewhat lowered. In this way one succeeds the Bragg condition to be met at two angles $\pm\Omega$ *outside* the plane of symmetry (therefore the name "Ω-Scan") which are registered during the rotation of the turntable, and from 2Ω the exact value of ε, and thus the exact orientation of the surface (i.e. the exact cutting angle) can be determined.

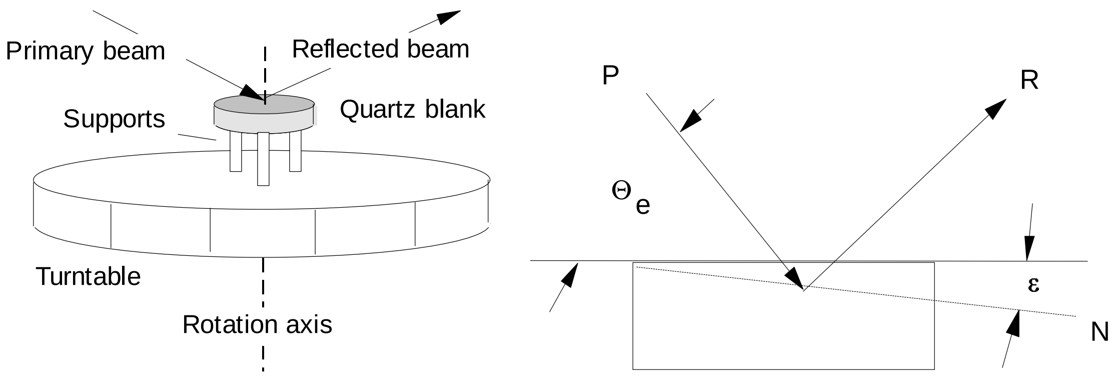


Fig. 13. Ω-Scan arrangement. Left-hand side: Turntable with 3-pin support holding the blank. Right hand side: The incident beam P is adjusted to hit the blank's surface at an angle $\Theta_e = \Theta_B + \varepsilon - \delta$ (ε=angle between surface and lattice plane N): due to the lowering at a small angle δ, no reflection R can occur within the sketched plane of symmetry. Only after rotating the turntable about two angles ±Ω two reflections appear and are registered during the continuous rotation of turntable + blank. From $\Omega = \Omega(\varepsilon)$ the exact value of ε will be calculated.

After the computer has calculated the ε value a sorter arm puts the blank into the corresponding one of 25 boxes, and in the meantime a feeder arm has brought the next blank into measuring position. The whole process takes about three seconds time and is completed during one rotation of the turntable. As an example Fig. 14 shows the result of such a cutting angle sorting of about 1000 blanks.

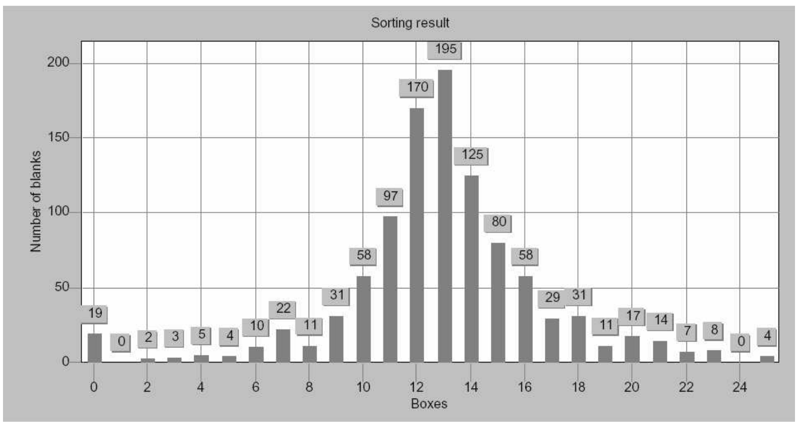


Fig. 14. Sorting result.

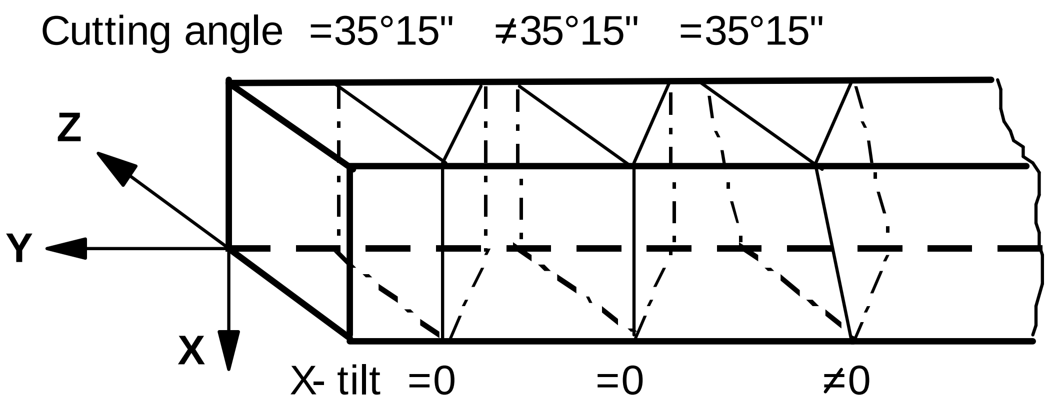


Fig. 15. Quartz Y-bar, orientation of the correct AT-cut (on the left hand side) and two different cutting errors.

There exist, however, serious problems resulting from mistakes during the cutting process. The following three errors can occur:

a) The cutted blanks are admittedly correctly oriented along the X-axis, see Fig. 15 (no "X-tilt"), as shown in the middle of Fig. 15, the cutting angle, however, differs from the correct value $\varphi - \varepsilon$, where φ is the angle bewteen lattice plane $(01\bar{1}1)$ and the XZ-plane $(0\bar{1}10)$. This kind of cutting error is easily detectable with the simple Ω-Scan method as described above.
b) The cutting angle is correct, the blank, however, is not cut parallel to the X-axis: "X-tilt $\neq 0$", also called "XX'-error", see Fig. 15, right hand side.
c) The bar itself is not correctly cut, the side planes are not parallel to the XY plane. The last two errors falsify Ω-Scan measurements, but have no influence, when an improved Ω-Scan method is applied.

The frequency/temperature stability is much more influenced by error a) than by error b), but in case of greater demands on the blank's stability (as they are usual nowadays for certain applications) also the X-miscutting error should be as small as possible. Bigger angles XX' enlarge the measured ε value: An X-tilt of 10 arcmin leads already to a measurement error of about 17 arcmin.

5.4. The "Improved Ω-Scan"

In order to eliminate the just mentioned problem, i.e. to determine the AT cutting angle and the XX' angle separately, the "Improved Ω- Scan" was developed [9]. It enables to measure four (instead of two) Bragg reflections at the two lattice planes $(01\bar{1}1)$ and $(02\bar{2}3)$. This could be realised using the same incident beam (the plane $(01\bar{1}1)$ is measured in second order reflection !), and also the same scintillation counter masked with a three hole lead sheet to screen other reflections and noise. The additional reflections on $(02\bar{2}3)$ are measured after a rotation of about 180° of the turntable, see Fig. 16.

Finally the measured and calculated XX' angle is used to correct the measured ε angle, and the result is a significant higher precision of the cutting angle determination: during 3 seconds measuring time (Ω= 360°) the precision reaches 3 arcsec (standard deviation).

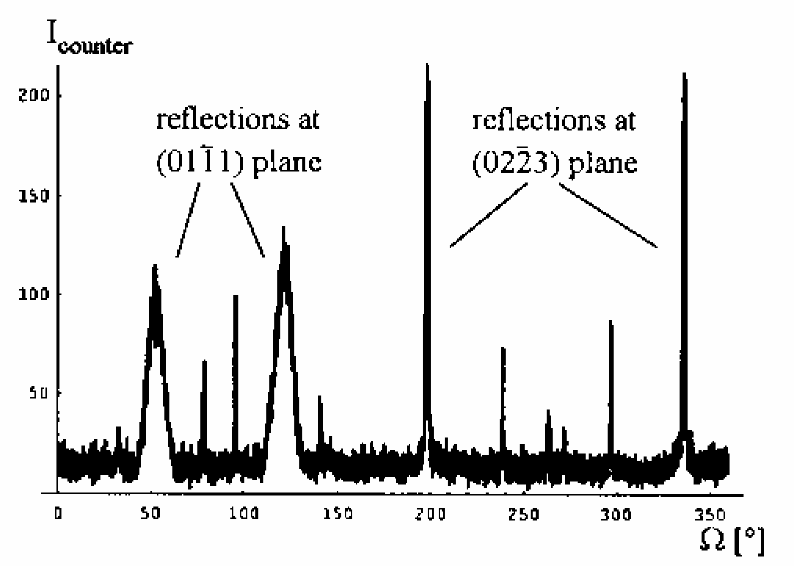


Fig. 16. The “Improved Ω-Scan“: Four reflections of type $(02\bar{2}2)$ and $(02\bar{2}3)$ measured during a 360° turn of the blank at lattice planes $(01\bar{1}1)$ and $(02\bar{2}3)$. From [9].

5.5. Other cuts with improved properties

As was already mentioned in Section 3 a number of cuts other than AT were introduced in recent years which offer improved properties concerning thermal stability, acceleration intensitivity etc. But since they do not occupy positions in the quartz lattice close to important lattice planes (as was the case with the AT cut) the adjustment of these improved blanks needs *two* rotations against prominent crystallographic axes, as was already shown in Fig. 10, and this is the reason why “doubly rotated blanks“ are cut at an increased manufacturing loss (with the consequence of increased production expenses).

Most of these doubly rotated blanks have a rectangular shape what them made suited for production control by the Theta-Scan, but the check of two angles needed two subsequent scans in rectangular directions, and this combination turned out to be difficult to handle.

Here the use of the Ω-Scan again brought a real advantage. The just discussed measurement of the XX’ correction had already considered two angles, even though against reflections from the same crystallographic zone. But principally the evaluation of two cutting angles of doubly rotated blanks was nothing more than a further step in the improvement of the Ω-Scan.

Useful pairs of reflections, appropriate for the measurement not only of SC-cut blanks, but also other doubly rotated cuts, have been found using a detailed computer program which considers all relevant quartz lattice planes. This enables not only to find out the most suited reflections but also to calculate the deviations of the measurements which have to be expected in any particular case [10]. In order to increase the precisions of the cutting angle determinations users usually apply “overlay measurements“, combining the results during four or eight rotations instead of one, see Fig. 17. Finally one arrives at standard deviations of about 2 arcsec.

So the measurement of doubly rotated-cut blanks is somewhat tricky in detail, but principally successfully feasible using the Ω-Scan method. The cutting process, however, remained to be a problematic procedure, mainly due to the fact that industry uses the AT-sawing equipment likewise for the production of SC (FC, IT, ...) doubly rotated blanks. Consequently the side faces of the bars must then be prepared in complicated positions relative to the XYZ quartz coordinates. That introduces additional sources for cutting errors. This was the reason to develop an X-ray controlled saw (already shortly mentioned in Section 3) which checks each cutting process separately. The cutting direction can be controlled by a computer. This saw offers two advantages: The use of the usual AT-bars with side faces parallel to the XY- and XZ-planes, and a high determinability of both cutting angles. As a disadvantage, the cutting occurs blank by blank, resulting in a cutting time of some minutes per blank.

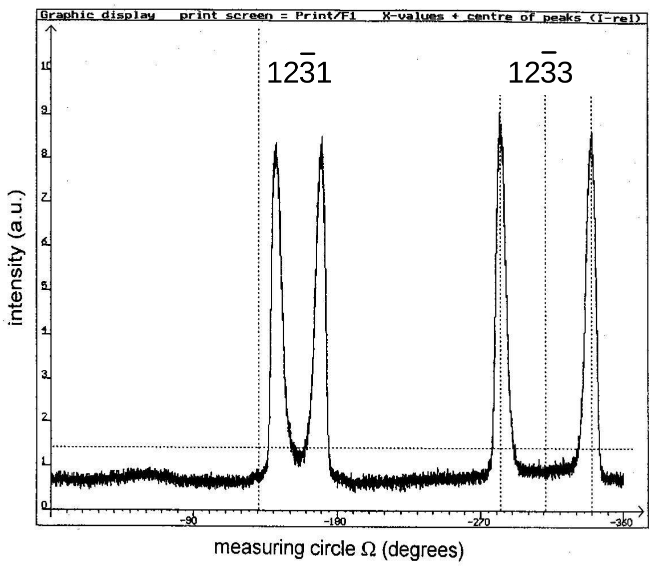


Fig. 17. SC-cut, reflections $12\bar{3}1$ and $12\bar{3}3$ measured with the Ω-Scan as average over eight 360° turns. - From [10].

6. Recent developments of technical equipment

The just described measuring method, the Ω-Scan, was capable in principle of delivering sufficiently exact results as long as no mechanical problems occured. As one of the most serious of such problems a slight wobbling of the blanks had been observed. At first glance the axle-bearing of the turntable was found to be the responsible component: even minute wobbling amplitudes in the range of less than 1 μm caused serious errors of the resulting values for the cutting angle. Although a mechanical adjustment arrangement was installed a sufficient correction of these (reproducible) errors could not be gained in that way.

There remained additionally unreproducible wobbling problems. They could be attributed to the influence of tiny quartz fragments and other dirt between blank and turntable. This was supported by REM (raster electron microscope) studies of quartz blank surfaces which had been treated in different ways [11].

Following a suggestion by J. R. Vig [12] a laser device had been installed [13] to eliminate both wobbling error sources by applying relevant corrections. The system uses the position of a laser beam after its reflection at the blank surface, measured with a CCD-camera, to calculate and consider the necessary correction of the final measuring results. In a very recent device even two lasers are used to check the positions of upper and lower surfaces of the blanks separately and to make sure that they are parallel to each other (see Fig. 18).

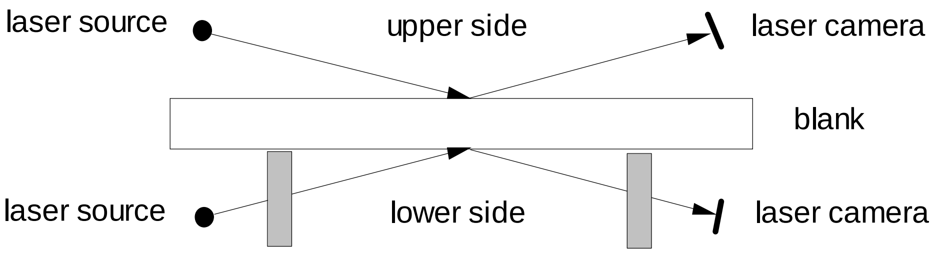


Fig. 18. The laser arrangement.

In another production line strip blanks are an intermediate aim. Here the starting point are larger wafers with sizes of e.g. 65 × 65 mm^2 and thicknesses of about 100 µm. These wafers are then cut into strips of suited sizes (e.g. 1 × 1 mm^2). Of course also this production process needs exact measurements of the cutting angle. Again the Omega-Scan method is successfully applied to its determination, which is nowadays split into two directions:

1) A more or less complete point-by-point checking across the surface of the whole wafer (at customer-preselected points, with minimal distances 1 mm in both directions), or
2) the sorting strip-by strip (using e.g. the "tandem" machine mentioned below). As is evident the second method is leading to a higher reliability, but at the expense of a much higher consumption of measuring time. Nevertheless both methods are currently in use.

A partial view of an AT-wafer sorting machine is shown in Fig. 19.

Fig. 19. Photograph of a wafer sorting machine. X X-ray tube, D detector, T turntable, P pick-up system, L laser camera, S_1 wafer sample held by lifter, S_2 samples stored in boxes (EFG, Berlin).

In order to increase the throughput of the blank sorting especially for the just mentioned mini-strip blanks a new system has been developed that uses two turntables (instead of one in the previous machines), see Fig. 20. A fast feeding and sorting system takes care the turntables not to have any intermediate breaks between the measurements. This has been made possible by still using only one X-ray tube.

With this new “tandem-machine“ the throughput could be increased by a factor of about 2.5 compared to the existing mono-sorters. With standard sorting accuracy 2500 blanks can now be measured per hour.

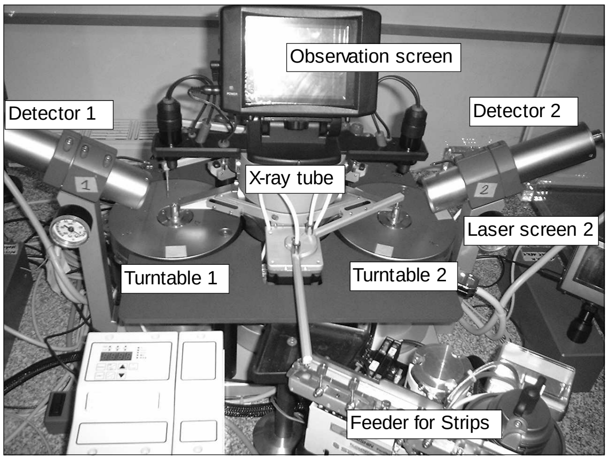


Fig. 20. X-ray bisorter (the “tandem-machine“) for strips down to 1×1 mm^2 and 20 μm thickness.

7. Applications of the Ω-Scan to materials other than quartz

7.1. Sapphire

Beside quartz, a material with a broad range of technical applications in future is sapphire, which already is used as a basic material for diodes and light emission parts.
The sapphire crystals are grown from the melt and drilled into ingots which are then sawed into wafers. These wafers are round and have a flat in a special crystallographic orientation.

The axis of the sapphire ingots is nearly [0001]. The corresponding lattice plane (0001) has to be adjusted to be parallel to the cutting plane of the sawing device. This is done by means of the Ω-Scan in the following way: Because of the trigonal symmetry the rotation of the ingot around its axis causes reflections of the type $hh\overline{2h}l$ or $h0\bar{h}l$ to appear threefold delivering three reflection pairs. The evaluation utilizes the positions of all these peaks. From that, two angular components (tilt angle and azimuth) for adjusting the ingot relative to the glueing device are calculated. The adjustment angle can be determined within some seconds of arc if this accuracy can be maintained by the sawing process.

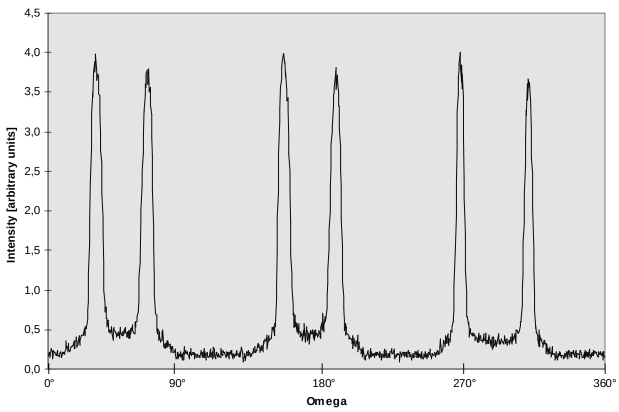


Fig. 20. Omega-Scan diagram of sapphire, three reflexions of type $(h0\bar{h}l)$, CuK_α radiation.

After sawing the ingot into wafers, these have to be controlled concerning the accuracy of the orientation. This can be performed using the same reflections as for the adjustment (see Fig. 20) and applying the laser correction for the determination of the exact angular position of the surface as described before for quartz blanks and wafers. In some cases it seems to be necessary to measure several points on the surface of the wafer. The Ω-Scan is suited to check the orientation in up to some hundred points.

7.2. Silicon carbide

Silicon carbide is industrially applied for similar purposes as sapphire. Wafers with the orientation [0001] and with deviations up to some degrees from [0001] are cut and are to be measured. However, for the polytypes with hexagonal symmetry, six symmetrically equivalent reflection pairs occur, as shown in Fig. 21. If there is a tilt of the crystal axis or of the surface, resp., of a few degrees, then some of the reflections can be superimposed or can vanish so that a rather sophisticated procedure is necessary to identify and to evaluate the reflections automatically.

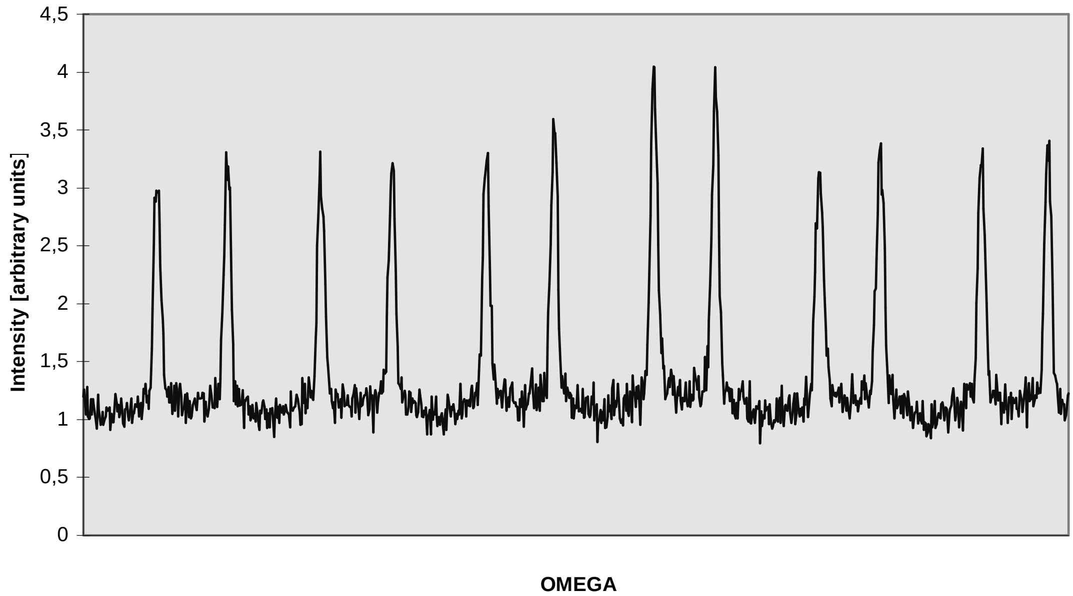


Fig. 21. Omega-Scan diagram of SiC Orientation [0001], reflections $(10\bar{1}12)$ and symmetrically equivalent ones, radiation CuK_{α} (measurements Figs. 20, 21: H. Berger).

8. Conclusions

For about a dozen years the above described Omega-Scan method has been applied to quartz helping industry to reduce the rate of rejected blanks or wafers from some 50% down to about 2%.

Recently this method has successfully been employed also to other crystalline materials which are of interest to industry. So one can conclude that the method should be suitable to apply it to arbitrary single crystals with arbitrary lattice geometry and symmetry. Suited reflections have to be chosen accordingly, e.g. equivalent reflections of one type for materials with high symmetry like sapphire or silicon carbide.

If necessary, a universal measuring device for the determination of the surface orientation (including laser correction) can be constructed, applicable for crystal material of arbitrary lattice geometry and symmetry. An adjusting apparatus preparing a succeeding cutting must be specified for the cutting device and procedure. Also in this case the Ω-Scan allows to be combined with such an apparatus fulfilling various demands.

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